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Canada Centre for Mineral and Energy Technology
Centre canadien de la technologie des minéraux et de l'énergie

ZINC-COPPER ORE RU-1: ITS CHARACTERIZATION AND PREPARATION FOR USE AS A CERTIFIED REFERENCE MATERIAL

G.H. Faye, W.S. Bowman and R. Sutarno

CANADIAN CERTIFIED REFERENCE MATERIALS PROJECT

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FOREWORD

The work described in this report contributes to the Canadian Certified Reference Materials Project (CCRMP). The CCRMP in turn contributes to the Utilization Activity (Quality Control sub-Activity) of CANMET's Minerals Research Program by producing mineralogical and metallurgical reference materials (RM's) for use in industrial, commercial and government laboratories in Canada.

The CCRMP was initiated in the early seventies in response to a demand from such laboratories for RM's that were not previously available. Many laboratories now work on their own behalf by willingly contributing analytical information which is ultimately used in the CCRMP to certify RM's.

Now that a relatively large number of reference ores and related materials have been made available, they are being used in a "feed-back" fashion to critically assess analytical methods that are essential for quality-control and research in Canadian enterprises.

R L Cunningham

R. L. Cunningham,
Chief

AVANT-PROPOS

Le travail qui est décrit dans le présent rapport apporte une contribution au Programme canadien des matériaux de référence certifiés (CCRMP). De son côté, le CCRMP collabore aux travaux de l'Activité d'utilisation (la sous-activité de surveillance de la qualité) du Programme de recherche sur les minéraux de CANMET en normalisant des matériaux minéralogiques et métallurgiques pour les différents laboratoires industriels, commerciaux et gouvernementaux au Canada.

Le CCRMP a été créé au début des années '70 pour répondre à la demande formulée par les différents laboratoires qui voulaient de tels matériaux de référence qui n'étaient pas disponibles auparavant. Ainsi, plusieurs laboratoires effectuent maintenant des travaux analytiques et par la suite lèguent volontairement les informations nécessaires au CCRMP pour certifier des matériaux de référence.

Maintenant qu'une quantité relativement abondante de minerais de référence et apparentés sont disponibles, on les utilise rétro-activement afin d'évaluer les méthodes analytiques employées par les compagnies canadiennes pour contrôler la qualité et faire de la recherche.

R. L. Cunningham,
Chef

CANMET REPORT 77-7

ZINC-COPPER ORE RU-1: ITS CHARACTERIZATION AND PREPARATION FOR USE AS A CERTIFIED REFERENCE MATERIAL

by

G.H. Faye, W. S. Bowman and R. Sutarno*

SYNOPSIS

As a facet of the Canadian Certified Reference Materials Project, a zinc-copper ore, RU-1, has been prepared as a compositional reference material. Approximately 300kg of raw ore was dry-ground to minus 74μ blended, tested for homogeneity by X-ray fluorescence and chemical methods, and bottled in 200-g units.

In a "free-choice" program for the certification of RU-1, 25 laboratories each provided analytical results for zinc, copper, iron and sulphur on each of two bottles of the ore. A statistical treatment of the data yielded recommended values for the four constituents which are: zinc - 2.24%, copper - 0.85%, iron - 24.4%, and sulphur - 21.7%.

Experiments have shown that RU-1, like many other sulphide-bearing materials, should be protected from undue exposure to air. In properly capped bottles, RU-1 is expected to have a shelf-life of at least five years. The stability of RU-1 will be monitored periodically at CANMET.

*Note: Major contributions to the certification of RU-1 were made by other members of the staff of the Mineral Sciences Laboratories and by laboratories in many other organizations (see p 2).

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INTRODUCTION

This report describes the characterization and preparation of samples of a zinc-copper ore, RU-1, for use as a certified compositional reference material. The work is a facet of the Canadian Certified Reference Materials Project (CCRMP) to certify materials that are representative mainly of Canadian ore deposits, and have potential value in conventional analytical or earth sciences laboratories. Certified reference ores and related materials issued previously in the CCRMP are described in a catalogue[1] that is available from the Canada Centre for Mineral and Energy Technology, Department of Energy, Mines and Resources, Ottawa, Canada.

RU-1 was chosen as a reference material because its relatively low zinc and high iron and sulphur contents are appreciably different from those in the base-metal ores previously certified in the CCRMP. The interlaboratory or round-robin approach was used to obtain analytical results for one or more of zinc, copper, iron and sulphur from 25 laboratories, which used analytical methods of their choice. As expected at the outset, most results for copper and zinc were by atomic absorption spectroscopy, whereas the majority of iron and sulphur results were obtained by titrimetry and gravimetry respectively.

NATURE AND PREPARATION OF RU-1

RU-1 was donated to the CCRMP in late 1973 and is from the Ruttan Mine of Sherritt Gordon Mines Limited, Lynn Lake, Manitoba. The mineralogical composition, the approximate chemical composition, and a particle-size analysis of RU-1 are given in Tables 1, 2, and 3 respectively.

In the spring of 1975, RU-1 was dry-ground, by ball-milling, to pass a minus 74 μ screen. The powdered material, weighing approximately 300 kg, was tumbled in a 570-ℓ conical blender for approximately 20 hours. Upon opening the blender the bulk material was sampled systematically and, by X-ray fluorescence and chemical analysis for zinc and copper at CANMET, RU-1 was found to be sufficiently homogeneous to be bottled in 200-g units and to be distributed in the interlaboratory certification program.

TABLE 1

Calculated mineralogical composition*

Mineral	wt %
pyrite	30.0
pyrrhotite	10.0
sphalerite	3.8
chalcopyrite	2.5
plagioclase	14.5
chlorite	12.7
amphibole	10.0
quartz	7.0
orthoclase	3.6
magnetite	1.0
goethite	1.0
mica	1.0
dolomite	1.0
siderite	0.8
calcite	0.4
Total	99.3

*Mineralogical composition determined by W. Petruk and R.G. Pinard, Physical Sciences Laboratory, CANMET.

TABLE 2

Results of approximate chemical and emission spectrographic analyses of RU-1

	Chemical[a] wt %	Emission spectrographic[b] wt %
Zn	2.24[c]	2.0
Cu	0.85[c]	0.85
Fe	24.41[c]	--
S	21.71[c]	--
Si	12.1	12
Al	4.3	4.3
Ca	2.8	3.4
Mg	3.3	3.3
Na	0.8	0.5
K	0.6	--
Mn	0.1	0.1
Ti	0.2	0.2
Ni	0.007	0.005
Cd	0.007	--
Zr	--	0.01
Sr	--	0.008
Co	--	0.01
Ba	--	0.009
Cr	--	0.009
C	0.3	--
H_2O (at 950°C)	1.7	--
Moisture (at 105°C)	0.3	--
Ag	7 ppm	--
Au	0.3 ppm	--

[a]Except for Zn, Cu, Fe and S, results are those provided by the Chemical Laboratory at CANMET.

[b]Results are means of five replicate analyses by direct reading emission spectrometry at Geological Survey of Canada.

[c]Recommended values from Table 8.

TABLE 3

Particle size analysis (Haultain infrasizer)

Particle size (μ)	wt %
+ 30	8
-30 + 15	25
-15 + 8	23
- 8	44

INTERLABORATORY PROGRAM FOR CERTIFICATION OF RU-1

The names of the laboratories that participated in the program to certify RU-1 are given below in alphabetical order. Each of these was arbitrarily assigned a code number so that analytical results could be recorded while preserving the anonymity of the laboratory. The code numbers bear no relation to the alphabetical order of the laboratory name.

Participating Laboratories

Bondar-Clegg and Company Limited,
Ottawa, Ontario.

Bondar-Clegg and Company Limited,
Vancouver, British Columbia.

British Columbia Department of Mines and
Petroleum Resources,
Victoria, British Columbia.

Canada Centre for Mineral and Energy
Technology, Mineral Sciences Laboratories
(four independent analysts),
Ottawa, Ontario.

Can Test Limited,
Vancouver, British Columbia.

Chemex Labs Limited,
North Vancouver, British Columbia.

Cominco Limited,
Trail, British Columbia.

Falconbridge Nickel Mines Limited,
Metallurgical Laboratories,
Thornhill, Ontario.

Geological Survey of Canada,
Central Laboratories and Administrative
Services, Ottawa, Ontario.

Hudson Bay Mining and Smelting Company Ltd.,
Flin Flon, Manitoba.

Inco Limited,
Analytical Services, Process Technology,
Copper Cliff, Ontario.

Lakefield Research of Canada, Limited,
Lakefield, Ontario.

LKAB,
Kiruna, Sweden.

Loring Laboratories Limited,
Calgary, Alberta.

Manitoba Department of Mines, Resources and
Environmental Management, Mines Branch,
Winnipeg, Manitoba.

Ministère des Richesses Naturelles,
Centre de Recherches Minérales,
Ste-Foy, Québec.

Ministry of Natural Resources,
Mineral Research Branch,
Toronto, Ontario.

Noranda Mines Limited,
Noranda, Quebec.

Sherritt Gordon Mines Limited,
Research and Development Division,
Fort Saskatchewan, Alberta.

Sherritt Gordon Mines Limited,
Mining and Milling Division,
Lynn Lake, Manitoba.

Thunder Bay Testing Limited,
Thunder Bay, Ontario.

United Keno Hill Mines Limited,
Elsa, Yukon.

The participating laboratories (or independent analysts at CANMET) received two randomly-selected bottles of RU-1, and were requested to determine total zinc, copper, iron and sulphur, in quintuplicate, in each bottle by methods of their choice. In cases where the participating organization provided results for a selected element by more than one method, each set of results was treated statistically as if it originated in a separate laboratory.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

All the analytical data obtained for zinc, copper, iron, and sulphur in RU-1 are presented in Tables 4(a) to 4(d) respectively. Table 5 correlates these results with the methods used and gives information on variations of particular methods, including sample decomposition. Tables 6(a) to 6(d) give the mean values and coefficients of variation for each set of results.

Detection of Outliers

To avoid the possible introduction of bias to the estimates of mean and variance for a particular element, results whose means differed by more than twice the overall standard deviation from the consensus value, and sets of results whose coefficients of variation were much larger than the norm were not used in the computations. These results are identified in Tables 4 and 6.

Of a total of 200 results for sulphur, 170 were by the conventional gravimetric method and 30 were by the combustion method. The three sets of combustion results are indicated in Figure 1(d) with the letter "C". Two of these are at the high end of the scale and are of relatively low precision; no doubt a contributing factor is that relatively small sample weights were used for the combustion method (Table 4(d)). Because of this, it was arbitrarily decided not to use these results in subsequent computations. It should be noted that the three laboratories which reported results by the combustion method also gave results by the conventional gravimetric method.

Confirmation of homogeneity using interlaboratory results

Using the t-test at the 5% significance level, a comparison was made of the reported results for both bottles from each set. In cases where the null hypothesis of no difference between bottles was rejected, the set is marked "REJECT" in Table 6; if the null hypothesis was accepted, the set is marked "A". From Table 6 it can be seen that there are rejects in 3 of 34 sets for zinc, 6 of 37 sets for copper, 8 of 25 sets for iron, and 3 of 20 sets for sulphur.

The degree of homogeneity of RU-1 is also illustrated in Figure 1, in which, for each set, the difference between the means of the results for the two bottles is plotted against the corresponding mean of the results for both bottles. The vertical bar represents the 95% confidence interval of the former. If a bar intersects the abscissa, the null hypothesis is accepted, i.e., there is no evidence of inhomogeneity between bottles for that set of results.

Because almost all of the sets of results contained exactly five replicates for each of two bottles, a two-way analysis of variance with nested design[2] was performed in order to resolve the within-set variance into between-bottle and within-bottle components. (Two copper sets for one bottle only were excluded from this computation along with the other outliers). The F-ratios of between-bottle to within-bottle mean squares were found to be 3.14, 1.78, 5.39, and 0.57 for zinc, copper, iron, and sulphur, respectively. (The critical value for the F distribution at 95% probability is about 1.5 for the degrees of freedom for this data). Only in the case of sulphur was the between-bottle inhomogeneity judged to be insignificant.

It is clear that bottle to bottle differences do exist. The largest component of variance, however, is between sets (laboratories). Therefore, because of its small magnitude, the between-bottle inhomogeneity should not be important in most applications of RU-1 as a reference material.

Estimation of consensus values and 95% confidence limits

Although the consensus values and variance of the means can be calculated from the two-way ANOVA, a one-way ANOVA technique was used in preference; this has the advantage that all results can be used, regardless of the number of replicates for each bottle or the number of bottles. For RU-1, this was necessary only in the case of copper. For zinc, iron, and sulphur, both the one-way and two-way ANOVA give identical estimates of the mean and its variance. In the one-way ANOVA, the data are assumed to fit the following model[2]:

$$x_{ij} = \mu + y_i + e_{ij}$$

where:

- x_{ij} = the j^{th} result reported in set i;
- μ = the true value that is estimated by the overall mean $\bar{x}_{..}$;
- y_i = the discrepancy between the mean of the results from set i and the true value; and
- e_{ij} = the discrepancy of x_{ij} from the means of the results from set i.

It is assumed in this analysis that both y_i and e_{ij} are normally distributed with means of zero and variances of ω^2 and σ^2, respectively. The significance of ω^2 can be detected by comparing the ratio of "between-set" mean squares to "within-set" mean squares with the F statistic at the 95% confidence level and with the appropriate degrees of freedom. The magnitude of ω^2 and σ^2 can be estimated from the ANOVA table.

Analysis of variance and expected mean squares for the one-way classification

Source of variance	Sums of squares	Degrees of freedom	Mean squares	E [Mean squares]
Between-sets	$\sum_i^k n_i (\bar{x}_{i.} - \bar{x}_{..})^2$	k-1	s_2^2	$\sigma^2 + \frac{1}{k-1}\left(\sum_i^k n_i - \frac{\sum_i^k n_i^2}{\sum_i^k n_i}\right)\omega^2$
Within-sets	$\sum_i^k \sum_j^{n_i} (x_{ij} - \bar{x}_{i.})^2$	$\sum_i^k n_i - k$	s_1^2	σ^2
Total	$\sum_i^k \sum_j^{n_i} (x_{ij} - \bar{x}_{..})^2$	$\sum_i^k n_i - 1$		

The consensus value, in the above model, can be estimated by the overall mean $\bar{x}_{..}$, thus:

$$\bar{x}_{..} = \frac{\sum_i^k \sum_j^{n_i} x_{ij}}{\sum_i^k n_i}$$

with the variance of the overall mean being given by:

$$V[\bar{x}_{..}] = \frac{\sum_i^k n_i^2}{\left(\sum_i^k n_i\right)^2}\omega^2 + \frac{1}{\sum_i^k n_i}\sigma^2$$

The 95% confidence limits for the overall mean are then given by:

$$\bar{x}_{..} \pm \left[t_{0.975\,(k-1)} \cdot \sqrt{V[\bar{x}_{..}]}\right]$$

where:

n_i = the number of results reported in set i;

k = the number of sets.

The above values and other statistics computed from the one-way ANOVA are presented in Table 7.

Certification factor

The certification factor[3] is a measure for evaluating the quality of reference materials issued by the CCRMP. It is computed from the following expression:

$$CF = 200\left[t_{0.975(k-1)} \cdot \sqrt{V[\bar{x}_{..}]}\right] / \bar{x}_{..} / \overline{cv}$$

where $\overline{cv}$ is the average of the within-set coefficients of variation and is given by:

$$\overline{cv} = \sum_i^k cv_i / k$$

The critical value of CF is 4. If a selected constituent has a CF greater than 4, the reference material is considered to be of unacceptable quality with respect to that constituent. Because the factors for RU-1 are all less than 4 (Table 7) the confidence of the estimate of the consensus values is as good as the average precision obtained by the contributors of the analytical results. Thus, these consensus values are accepted as recommended values for RU-1. They are listed along with their 95% confidence limits in Table 8.

DISCUSSION

Analytical methods

Table 5 gives an outline of the variations of the analytical methods used for the four selected elements in RU-1. Where applicable, methods of decomposition are given because these could, potentially, lead to biased results if inappropriately chosen. However, where a substantial number of results are reported, the mean values indicate that there is no significant difference between either the overall nature of the methods or the methods of sample decomposition.

Stability of RU-1

RU-1 contains appreciable concentrations of pyrite and pyrrhotite, minerals that oxidize readily under relatively moderate temperatures in the presence of water. To determine how RU-1 would be affected by extreme storage conditions or direct exposure to a laboratory atmosphere, samples were subjected to temperatures of 25° and 52°C and relative humidities of 58 and 70% respectively for periods of up to 57 days.

Table 9 shows that after 57 days at ambient temperature and 58% relative humidity, RU-1 increases in weight by 1.2%. However, less than half of this gain can be attributed to oxidation---the major part being due to adsorbed moisture. At 52°C and 70% relative humidity, there is rapid gain in weight after short periods of exposure, and from Table 9 it can be inferred that from one third to one half of this change is related to the formation of elemental sulphur, which is known to be a major oxidation product of pyrrhotite. Because pyrrhotite comprises only $\sim$ 10% by weight of RU-1, (Table 1) it is evident that a substantial fraction of this mineral is oxidized after 35 days exposure under the extreme conditions of 52°C and 70% relative humidity.

Although RU-1, like other sulphide-bearing materials, is potentially prone to oxidation if normal care is not exercised in protecting it from undue exposure to the atmosphere, the extent of change of RU-1 kept in properly capped bottles is expected to be sufficiently small that its useful life as a reference material should be at least five years. This is supported by the observation that analytical results for RU-1 were acquired in the interlaboratory round-robin over a period of 14 months and no "time effect" was indicated by a statistical analysis of the data. It is to be emphasized, however, that the recommended values given in Table 8 are those that pertain to RU-1 in December 1976. The stability of RU-1 will be monitored periodically at CANMET and users will be notified if oxidation effects are detected.

REFERENCES

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2. BROWNLESS, K.A., Statistical theory and methodology in sciences; John Wiley and Sons, Inc., New York; 1961.

3. SUTARNO, R. and FAYE, G.H., A measure for assessing certified reference ores and related materials, Talanta; v. 22, pp 676-681; 1975.

TABLE 4(a)

Zinc results for RU-1

ZINC (WEIGHT PERCENT)

											SAMPLE WT, G
LAB- 1 (A.A.)	2.250	2.250	2.270	2.260	2.260	2.280	2.280	2.280	2.260	2.260	1.0
LAB- 2 (A.A.)	2.210	2.220	2.220	2.240	2.220	2.230	2.230	2.200	2.220	2.230	0.5
LAB- 2 (VOL.)	2.290	2.280	2.290	2.270	2.290	2.270	2.270	2.280	2.290	2.280	0.5
LAB- 3 (POLAR.)	2.210	2.210	2.190	2.190	2.260	2.220	2.220	2.200	2.240	2.240	0.5
LAB- 4 (A.A.)	2.270	2.270	2.270	2.270	2.260	2.270	2.280	2.270	2.280	2.270	2.0
LAB- 4 (VOL.)	2.250	2.270	2.260	2.270	2.270	2.250	2.250	2.270	2.240	2.260	2.0
* LAB- 5 (A.A.)	2.000	2.100	2.000	2.100	2.100	2.000	2.100	2.000	2.100	2.000	0.2
* LAB- 5 (VOL.)	2.000	2.000	2.100	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.0
LAB- 6 (A.A.)	2.280	2.270	2.300	2.260	2.280	2.280	2.280	2.270	2.280	2.300	0.5
LAB- 6 (POLAR.)	2.320	2.340	2.340	2.320	2.350	2.240	2.240	2.270	2.250	2.270	1.0
LAB- 7 (A.A.)	2.216	2.220	2.230	2.225	2.229	2.220	2.230	2.222	2.230	2.234	1.0
LAB- 8 (A.A.)	2.200	2.210	2.200	2.210	2.210	2.190	2.200	2.200	2.210	2.200	0.5
LAB-10 (A.A.)	2.320	2.270	2.340	2.300	2.360	2.280	2.310	2.330	2.330	2.320	0.5
LAB-10 (VOL.)	2.260	2.310	2.280	2.260	2.250	2.270	2.320	2.300	2.280	2.260	1.0
LAB-11 (A.A.)	2.240	2.230	2.250	2.230	2.250	2.240	2.250	2.240	2.250	2.250	0.5
LAB-12 (A.A.)	2.120	2.150	2.160	2.150	2.180	2.170	2.120	2.160	2.160	2.150	0.5
LAB-12 (VOL.)	2.200	2.210	2.210	2.200	2.120	2.200	2.200	2.220	2.220	2.210	2.0
LAB-13 (A.A.)	2.264	2.274	2.279	2.286	2.287	2.274	2.273	2.284	2.277	2.277	1.0
LAB-14 (A.A.)	2.300	2.273	2.257	2.293	2.267	2.266	2.287	2.273	2.254	2.265	1.0
LAB-15 (A.A.)	2.150	2.150	2.160	2.140	2.140	2.140	2.150	2.150	2.140	2.150	0.5
LAB-15 (VOL.)	2.180	2.160	2.160	2.170	2.170	2.150	2.180	2.170	2.150	2.160	2.0
LAB-16 (A.A.)	2.250	2.270	2.270	2.230	2.230	2.240	2.240	2.200	2.210	2.210	0.25
LAB-18 (A.A.)	2.230	2.230	2.250	2.230	2.230	2.250	2.230	2.230	2.230	2.230	0.5
LAB-19 (A.A.)	2.260	2.200	2.220	2.200	2.200	2.260	2.200	2.220	2.210	2.190	1.0
LAB-19 (VOL.)	2.160	2.150	2.180	2.150	2.170	2.160	2.150	2.170	2.170	2.170	2.0
LAB-20 (A.A.)	2.220	2.260	2.270	2.260	2.260	2.220	2.260	2.230	2.240	2.260	0.2
LAB-22 (A.A.-1)	2.240	2.220	2.240	2.240	2.240	2.240	2.240	2.240	2.230	2.240	0.5
LAB-22 (A.A.-2)	2.220	2.220	2.220	2.210	2.220	2.230	2.230	2.230	2.220	2.220	0.5
LAB-25 (A.A.)	2.220	2.240	2.250	2.240	2.250	2.230	2.260	2.230	2.220	2.240	1.0
* LAB-26 (SPECTR.1)	2.100	2.330	2.310	2.050	2.160	2.250	1.990	1.900	2.040	1.800	0.6
* LAB-26 (SPECTR.2)	1.840	2.160	2.280	1.950	1.890	1.940	2.110	2.360	1.910	1.990	0.1
LAB-27 (A.A.)	2.220	2.240	2.250	2.200	2.200	2.240	2.220	2.220	2.250	2.240	0.25
LAB-28 (A.A.)	2.250	2.250	2.250	2.250	2.250	2.250	2.240	2.250	2.250	2.260	1.0
LAB-29 (A.A.)	2.260	2.260	2.310	2.300	2.270	2.260	2.250	2.300	2.280	2.260	0.2

*Sets judged to be outliers.

TABLE 4(b)

Copper results for RU-1

COPPER (WEIGHT PERCENT)

											SAMPLF WT. G
LAB- 1 (A.A.)	.873	.876	.884	.882	.881	.883	.878	.880	.869	.881	1.0
LAB- 2 (A.A.-1)	.868	.886	.868	.877	.877	.860	.873	.868	.873	.868	0.5
LAB- 2 (A.A.-2)	.889	.891	.890	.886	.890	.919	.911	.897	.913	.897	0.5
LAB- 2 (COLOR.)	.889	.879	.898	.900	.879	.853	.863	.879	.868	.874	0.5
LAB- 3 (POLAR.)	.838	.847	.842	.833	.857	.848	.843	.845	.852	.846	0.5
LAB- 4 (A.A.)	.856	.860	.856	.854	.855	.855	.860	.853	.855	.851	2.0
LAB- 4 (VOL.)	.850	.850	.850	.860	.860	.850	.860	.860	.860	.860	2.0
LAB- 5 (A.A.)	.820	.830	.820	.820	.830	.810	.810	.800	.800	.800	0.2
LAB- 6 (A.A.)	.830	.820	.840	.790	.850	.830	.820	.830	.790	.840	0.5
LAB- 6 (POLAR.)	.840	.850	.870	.850	.850	.860	.850	.850	.850	.830	1.0
LAB- 7 (A.A.)	.862	.858	.860	.864	.856	.858	.864	.860	.860	.862	1.0
LAB- 8 (A.A.)	.850	.860	.860	.850	.850	.860	.860	.850	.860	.860	0.5
LAB-10 (A.A.)	.860	.840	.820	.840	.870	.820	.890	.880	.830	.850	1.0
LAB-10 (VOL.)	.830	.840	.830	.870	.860	.840	.830	.880	.830	.860	1.0
LAB-11 (A.A.)	.860	.850	.850	.855	.855	.860	.855	.860	.860	.860	0.5
LAB-12 (A.A.-1)	.834	.834	.834	.834	.836	.834	.834	.840	.840	.847	0.5
LAB-12 (A.A.-2)	.870	.854	.870	.872	.870	.850	.866	.862	.858	.860	1.0
LAB-12 (VOL.)	.838	.850	.838	.850	.838	.838	.838	.838	.850	.850	2.0
LAB-13 (A.A.)	.864	.871	.868	.877	.872	.875	.870	.874	.874	.870	1.0
LAB-13 (VOL.)	.865	.870	.858	.882	.868	.878	.868	.868	.871	.876	1.0
LAB-14 (A.A.)	.845	.845	.847	.855	.843						1.0
LAB-14 (COLOR.)	.868	.855	.834	.838	.851						0.5
LAB-15 (A.A.)	.846	.848	.850	.848	.846	.850	.848	.852	.852	.848	0.5
LAB-16 (A.A.)	.876	.860	.860	.840	.845	.856	.852	.856	.855	.860	0.25
LAB-18 (A.A.)	.850	.850	.840	.850	.840	.870	.850	.850	.850	.860	0.5
LAB-19 (A.A.)	.840	.860	.850	.840	.860	.860	.860	.850	.850	.860	1.0
LAB-19 (VOL.)	.870	.870	.870	.870	.870	.870	.870	.870	.870	.880	2.5
LAB-20 (A.A.)	.843	.835	.840	.845	.836	.835	.843	.836	.836	.833	0.2
LAB-22 (A.A.)	.829	.834	.840	.838	.826	.836	.838	.825	.842	.828	0.5
LAB-22 (VOL.)	.814	.834	.834	.834	.834	.834	.834	.827	.834	.820	2.0
LAB-24 (X.R.F.)	.895	.896	.904	.875	.881	.875	.874	.876	.876	.875	**
LAB-25 (A.A.)	.850	.830	.830	.830	.830	.850	.830	.830	.820	.830	1.0
* LAB-26 (SPECTR.1)	.940	.940	.900	.970	.940	.940	.860	.900	.910	.860	0.6
* LAB-26 (SPECTR.2)	.769	.889	.907	.868	.808	.880	.908	.860	.757	.840	0.1
LAB-27 (A.A.)	.860	.850	.880	.860	.880	.890	.860	.870	.880	.890	0.25
LAB-28 (A.A.)	.853	.851	.849	.852	.849	.850	.848	.850	.844	.843	1.0
LAB-29 (A.A.)	.850	.850	.860	.860	.850	.860	.860	.850	.860	.850	0.1

*Sets judged to be outliers.

**5.0 g pellet

TABLE 4(c)

Iron results for RU-1

IRON (WEIGHT PERCENT)

										SAMPLE WT. G
LAB- 1 (VOL.)	24.60	24.50	24.60	24.60	24.60	24.60	24.60	24.60	24.60	24.60	0.5
LAB- 2 (VOL.)	24.48	24.43	24.43	24.37	24.43	24.48	24.37	24.37	24.48	24.43	0.5
LAB- 3 (VOL.)	24.43	24.44	24.42	24.44	24.44	24.43	24.42	24.43	24.45	24.44	0.5
LAB- 4 (VOL.)	24.43	24.38	24.40	24.35	24.35	24.35	24.38	24.35	24.33	24.35	0.5
LAB- 5 (VOL.)	24.30	24.30	24.30	24.30	24.30	24.30	24.30	24.30	24.30	24.20	0.5
*LAB- 6 (VOL.)	24.00	23.90	24.00	24.00	24.00	23.90	24.00	24.00	24.00	24.00	0.5
LAB- 7 (VOL.)	24.31	24.41	24.30	24.33	24.31	24.42	24.43	24.43	24.40	24.40	0.5
LAB- 8 (VOL.)	24.18	24.13	24.21	24.24	24.18	24.18	24.13	24.27	24.24	24.21	1.0
LAB-10 (VOL.)	24.74	24.72	24.74	24.70	24.60	24.72	24.70	24.59	24.69	24.67	1.0
LAB-11 (VOL.)	24.31	24.30	24.30	24.36	24.33	24.21	24.19	24.28	24.17	24.18	1.0
LAB-12 (VOL.)	24.65	24.75	24.75	24.65	24.65	24.65	24.65	24.65	24.65	24.65	0.25
LAB-13 (A.A.)	24.16	24.04	24.19	24.32	24.22	24.27	24.29	24.40	24.55	24.38	1.0
LAB-14 (VOL.)	24.24	24.16	24.23	24.10	24.11	24.30	24.21	24.18	24.30	24.34	0.5
LAB-15 (VOL.)	24.75	24.75	24.70	24.80	24.75	24.75	24.75	24.70	24.75	24.80	0.5
LAB-16 (VOL.)	24.27	24.31	24.33	24.32	24.35	24.23	24.28	24.36	24.25	24.29	0.4
LAB-18 (VOL.)	24.39	24.44	24.44	24.44	24.39	24.44	24.44	24.39	24.44	24.44	0.5
LAB-19 (VOL.)	24.44	24.44	24.42	24.42	24.44	24.42	24.42	24.42	24.40	24.44	0.5
LAB-20 (VOL.)	24.29	24.30	24.24	24.32	24.29	24.32	24.28	24.30	24.20	24.28	0.5
LAB-22 (VOL.)	24.40	24.55	24.40	24.55	24.50	24.50	24.35	24.55	24.50	24.45	0.5
LAB-22 (A.A.)	24.45	24.35	24.25	24.25	24.35	24.35	24.35	24.25	24.45	24.35	0.5
LAB-25 (VOL.)	24.40	24.50	24.60	24.50	24.40	24.20	24.10	24.20	24.40	24.50	0.4
LAB-27 (A.A.)	24.60	24.60	24.60	24.60	24.60	24.70	24.60	24.80	24.60	24.60	0.25
LAB-27 (VOL.)	24.36	24.22	24.36	24.22	24.22	24.22	24.08	23.94	24.22	24.08	0.5
LAB-27 (VOL.-P.)	24.27	24.27	24.32	24.27	24.38	24.10	24.16	24.13	24.27	24.05	0.5
LAB-29 (VOL.)	24.47	24.42	24.45	24.45	24.47	24.57	24.52	24.57	24.54	24.52	1.0

*Set judged to be outlier.

TABLE 4(d)

Sulphur results for RU-1

SULPHUR (WEIGHT PERCENT)

										SAMPLF WT. G
LAB- 1 (GRAV.)	21.80	22.10	21.70	22.30	22.20	21.70	21.70	22.20	22.30	22.20	0.5
LAB- 2 (GRAV.)	21.50	21.59	21.53	21.51	21.59	21.51	21.51	21.59	21.49	21.50	0.5
LAB- 3 (GRAV.)	21.56	21.61	21.54	21.54	21.54	21.54	21.51	21.53	21.51	21.55	0.3
LAB- 4 (GRAV.)	21.47	21.49	21.37	21.44	21.46	21.31	21.39	21.47	21.48	21.44	0.5
LAB- 5 (GRAV.)	21.61	21.67	21.64	21.66	21.59	21.67	21.62	21.59	21.67	21.67	0.5
LAB- 6 (GRAV.)	22.15	22.19	22.16	22.14	22.18	22.10	22.13	22.07	22.07	22.09	0.5
LAB- 7 (GRAV.)	21.60	21.54	21.60	21.56	21.48	21.62	21.64	21.56	21.50	21.49	1.0
LAB- 8 (GRAV.)	21.10	21.30	21.21	21.23	21.17	21.18	21.26	21.15	21.14	21.32	0.5
LAB-11 (GRAV.)	21.62	21.56	21.57	21.47	21.70	21.64	21.54	21.67	21.54	21.68	0.4
LAB-12 (GRAV.)	21.93	21.87	21.82	21.83	21.82	21.80	21.78	21.78	21.75	21.86	0.5
*LAB-12 (COMB.)	22.42	22.12	22.39	22.45	22.45	22.20	21.95	22.23	21.69	22.23	0.01
LAB-14 (GRAV.)	21.89	21.64	21.69	21.63	21.61	21.52	21.71	21.77	21.52	21.38	0.2
LAB-16 (GRAV.)	21.29	21.33	21.28	21.35	21.25	21.30	21.28	21.31	21.29	21.37	0.4
LAB-18 (GRAV.)	21.71	21.60	21.59	21.59	21.60	21.72	21.67	21.67	21.56	21.67	0.5
*LAB-19 (GRAV.)	22.30	22.30	22.30	22.10	22.10	22.10	22.30	22.30	22.20	22.20	0.25
*LAB-19 (COMB.)	22.40	22.40	22.20	22.40	22.40	22.40	22.00	22.40	22.40	22.00	0.05
LAB-22 (GRAV.)	21.64	21.42	21.53	21.69	21.64	21.50	21.50	21.58	21.64	21.64	0.5
*LAB-22 (COMB.)	21.60	21.60	21.60	21.70	21.70	21.70	21.70	21.80	21.70	21.60	0.1
LAB-25 (GRAV.)	21.60	21.57	21.68	21.50	21.73	21.66	21.67	21.59	21.59	21.59	0.5
LAB-27 (GRAV.)	21.58	21.61	21.59	21.50	21.54	21.65	21.58	21.68	21.67	21.61	0.5

*Sets judged to be outliers.

TABLE 5

Outline of analytical methods

	Decomposition	No. of laboratories	No. of results	Mean, wt %
Zinc				
Atomic absorption	m.a.* including HF	16	160(excluding 10 outliers)	2.23
	m.a. no HF	4	40	2.26
	not known	2	20	2.24
Volumetric, ferrocyanide	m.a. including HF	4	30(excluding 10 outliers)	2.18
, EDTA	m.a. (some with no HF)	3	30	2.27
Polarographic	m.a. including HF	2	20	2.26
Copper				
Atomic absorption	m.a. including HF	16	175	0.85
	m.a. no HF	4	40	0.85
	not known	2	20	0.85
Volumetric, thiosulphate	m.a. (some with no HF)	6	60	0.85
Absorptiometric	m.a. (some with no HF)	2	15	0.87
Polarographic	m.a. including HF	2	20	0.85
XRF		1	10	0.88
Iron				
Volumetric, dichromate	m.a. including HF	9	90	24.50
, dichromate	caustic fusion in Zr or Ni crucible	8	80(excluding 10 outliers)	24.34
, permanganate	m.a. including HF	1	10	24.20
, ceric amm. sulphate	caustic fusion	1	10	24.29
	not known	2	20	24.36
Atomic absorption	m.a. including HF	2	20	24.27
	caustic fusion	1	10	24.28
Sulphur				
Gravimetric, ($BaSO_4$)	m.a. no HF	9	90	21.73
	caustic fusion	7	70	21.55
, combustion		3	30	22.06

* m.a. - mixed acids, e.g. (HCl, HNO_3, $HClO_4$)

TABLE 6(a)

Laboratory means, coefficients of variation, and summary of t-test on between-bottle zinc results for RU-1

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(A.A.)	5	2.2580	.0084	5	2.2720	.0110	A	10	2.2650	.0118	.52
LAB- 2	(A.A.)	5	2.2220	.0110	5	2.2220	.0130	A	10	2.2220	.0114	.51
LAB- 2	(VOL.)	5	2.2840	.0089	5	2.2780	.0084	A	10	2.2810	.0088	.38
LAB- 3	(POLAR.)	5	2.2120	.0286	5	2.2240	.0167	A	10	2.2180	.0230	1.04
LAB- 4	(A.A.)	5	2.2680	.0045	5	2.2740	.0055	A	10	2.2710	.0057	.25
LAB- 4	(VOL.)	5	2.2640	.0089	5	2.2540	.0114	A	10	2.2590	.0110	.49
*LAB- 5	(A.A.)	5	2.0600	.0548	5	2.0400	.0548	A	10	2.0500	.0527	2.57
*LAB- 5	(VOL.)	5	2.0200	.0447	5	2.0000	0.0000	A	10	2.0100	.0316	1.57
LAB- 6	(A.A.)	5	2.2780	.0148	5	2.2820	.0110	A	10	2.2800	.0125	.55
LAB- 6	(POLAR.)	5	2.3340	.0134	5	2.2540	.0152	REJECT	10	2.2940	.0443	1.93
LAB- 7	(A.A.)	5	2.2240	.0060	5	2.2272	.0059	A	10	2.2256	.0059	.26
LAB- 8	(A.A.)	5	2.2060	.0055	5	2.2000	.0071	A	10	2.2030	.0067	.31
LAB-10	(A.A.)	5	2.3180	.0349	5	2.3140	.0207	A	10	2.3160	.0272	1.17
LAB-10	(VOL.)	5	2.2720	.0239	5	2.2860	.0241	A	10	2.2790	.0238	1.04
LAB-11	(A.A.)	5	2.2400	.0100	5	2.2460	.0055	A	10	2.2430	.0082	.37
LAB-12	(A.A.)	5	2.1520	.0217	5	2.1520	.0192	A	10	2.1520	.0193	.90
LAB-12	(VOL.)	5	2.1880	.0383	5	2.2100	.0100	A	10	2.1990	.0288	1.31
LAB-13	(A.A.)	5	2.2780	.0095	5	2.2770	.0043	A	10	2.2775	.0069	.31
LAB-14	(A.A.)	5	2.2780	.0180	5	2.2690	.0121	A	10	2.2735	.0152	.67
LAB-15	(A.A.)	5	2.1480	.0084	5	2.1460	.0055	A	10	2.1470	.0067	.31
LAB-15	(VOL.)	5	2.1680	.0084	5	2.1620	.0130	A	10	2.1650	.0108	.50
LAB-16	(A.A.)	5	2.2500	.0200	5	2.2200	.0187	REJECT	10	2.2350	.0242	1.08
LAB-18	(A.A.)	5	2.2340	.0089	5	2.2340	.0089	A	10	2.2340	.0084	.38
LAB-19	(A.A.)	5	2.2160	.0261	5	2.2160	.0270	A	10	2.2160	.0250	1.13
LAB-19	(VOL.)	5	2.1620	.0130	5	2.1640	.0089	A	10	2.1630	.0106	.49
LAB-20	(A.A.)	5	2.2540	.0195	5	2.2420	.0179	A	10	2.2480	.0187	.83
LAB-22	(A.A.-1)	5	2.2360	.0089	5	2.2380	.0045	A	10	2.2370	.0067	.30
LAB-22	(A.A.-2)	5	2.2180	.0045	5	2.2260	.0055	REJECT	10	2.2220	.0063	.28
LAB-25	(A.A.)	5	2.2400	.0122	5	2.2360	.0152	A	10	2.2380	.0132	.59
*LAB-26	(SPECTR.1)	5	2.1900	.1251	5	1.9960	.1689	A	10	2.0930	.1735	8.29
*LAB-26	(SPECTR.2)	5	2.0240	.1880	5	2.0620	.1832	A	10	2.0430	.1761	8.62
LAB-27	(A.A.)	5	2.2220	.0228	5	2.2340	.0134	A	10	2.2280	.0187	.84
LAB-28	(A.A.)	5	2.2500	0.0000	5	2.2500	.0071	A	10	2.2500	.0047	.21
LAB-29	(A.A.)	5	2.2800	.0235	5	2.2700	.0200	A	10	2.2750	.0212	.93
								TOTAL	340	2.2151	.0854	3.86

*Sets judged to be outliers.

TABLE 6(b)

Laboratory means, coefficients of variation, and summary of t-test on between-bottle copper results for RU-1

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(A.A.)	5	.8792	.0045	5	.8782	.0054	A	10	.8787	.0048	.54
LAB- 2	(A.A.-1)	5	.8752	.0075	5	.8684	.0053	A	10	.8718	.0071	.82
LAB- 2	(A.A.-2)	5	.8892	.0019	5	.9074	.0099	REJECT	10	.8983	.0117	1.31
LAB- 2	(COLOR.)	5	.8890	.0100	5	.8674	.0101	REJECT	10	.8782	.0148	1.69
LAB- 3	(POLAR.)	5	.8434	.0092	5	.8468	.0034	A	10	.8451	.0068	.80
LAB- 4	(A.A.)	5	.8562	.0023	5	.8548	.0033	A	10	.8555	.0028	.33
LAB- 4	(VOL.)	5	.8540	.0055	5	.8580	.0045	A	10	.8560	.0052	.60
LAB- 5	(A.A.)	5	.8240	.0055	5	.8040	.0055	REJECT	10	.8140	.0117	1.44
LAB- 6	(A.A.)	5	.8260	.0230	5	.8220	.0192	A	10	.8240	.0201	2.44
LAB- 6	(POLAR.)	5	.8520	.0110	5	.8480	.0110	A	10	.8500	.0105	1.24
LAB- 7	(A.A.)	5	.8600	.0032	5	.8608	.0023	A	10	.8604	.0026	.31
LAB- 8	(A.A.)	5	.8540	.0055	5	.8580	.0045	A	10	.8560	.0052	.60
LAB-10	(A.A.)	5	.8460	.0195	5	.8540	.0305	A	10	.8500	.0245	2.88
LAB-10	(VOL.)	5	.8460	.0182	5	.8480	.0217	A	10	.8470	.0189	2.23
LAB-11	(A.A.)	5	.8540	.0042	5	.8590	.0022	REJECT	10	.8565	.0041	.48
LAB-12	(A.A.-1)	5	.8344	.0009	5	.8390	.0054	A	10	.8367	.0044	.52
LAB-12	(A.A.-2)	5	.8672	.0074	5	.8592	.0059	A	10	.8632	.0076	.88
LAB-12	(VOL.)	5	.8428	.0066	5	.8428	.0066	A	10	.8428	.0062	.74
LAB-13	(A.A.)	5	.8704	.0048	5	.8726	.0024	A	10	.8715	.0038	.43
LAB-13	(VOL.)	5	.8686	.0088	5	.8722	.0046	A	10	.8704	.0069	.79
LAB-14	(A.A.)	THERE IS ONLY 1 BOTTLE							5	.8470	.0047	.55
LAB-14	(COLOR.)	THERE IS ONLY 1 BOTTLE							5	.8492	.0137	1.61
LAB-15	(A.A.)	5	.8476	.0017	5	.8500	.0020	A	10	.8488	.0021	.25
LAB-16	(A.A.)	5	.8562	.0142	5	.8558	.0029	A	10	.8560	.0097	1.13
LAB-18	(A.A.)	5	.8460	.0055	5	.8560	.0089	A	10	.8510	.0088	1.03
LAB-19	(A.A.)	5	.8500	.0100	5	.8560	.0055	A	10	.8530	.0082	.97
LAB-19	(VOL.)	5	.8700	.0000	5	.8720	.0045	A	10	.8710	.0032	.36
LAB-20	(A.A.)	5	.8398	.0043	5	.8366	.0038	A	10	.8382	.0042	.50
LAB-22	(A.A.)	5	.8334	.0059	5	.8338	.0071	A	10	.8336	.0061	.74
LAB-22	(VOL.)	5	.8300	.0089	5	.8298	.0063	A	10	.8299	.0073	.88
LAB-24	(X.R.F.)	5	.8902	.0119	5	.8752	.0008	REJECT	10	.8827	.0112	1.27
LAB-25	(A.A.)	5	.8340	.0089	5	.8320	.0110	A	10	.8330	.0095	1.14
* LAB-26	(SPECTR.1)	5	.9380	.0249	5	.8940	.0344	REJECT	10	.9160	.0366	3.99
* LAB-26	(SPECTR.2)	5	.8482	.0579	5	.8490	.0572	A	10	.8486	.0543	6.40
LAB-27	(A.A.)	5	.8660	.0134	5	.8780	.0130	A	10	.8720	.0140	1.60
LAB-28	(A.A.)	5	.8508	.0018	5	.8470	.0033	A	10	.8489	.0032	.38
LAB-29	(A.A.)	5	.8540	.0055	5	.8560	.0055	A	10	.8550	.0053	.62
								TOTAL	360	.8559	.0242	2.83

*Sets judged to be outliers.

TABLE 6(c)

Laboratory means, coefficients of variation, and summary of t-test on between-bottle iron results for RU-1

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(VOL.)	5	24.5800	.0447	5	24.6000	.0000	A	10	24.5900	.0316	.13
LAB- 2	(VOL.)	5	24.4280	.0390	5	24.4260	.0550	A	10	24.4270	.0450	.18
LAB- 3	(VOL.)	5	24.4340	.0089	5	24.4340	.0114	A	10	24.4340	.0097	.04
LAB- 4	(VOL.)	5	24.3820	.0342	5	24.3520	.0179	A	10	24.3670	.0302	.12
LAB- 5	(VOL.)	5	24.3000	.0000	5	24.2800	.0447	A	10	24.2900	.0316	.13
*LAB- 6	(VOL.)	5	23.9800	.0447	5	23.9800	.0447	A	10	23.9800	.0422	.18
LAB- 7	(VOL.)	5	24.3320	.0449	5	24.4160	.0152	REJECT	10	24.3740	.0544	.22
LAB- 8	(VOL.)	5	24.1880	.0409	5	24.2060	.0541	A	10	24.1970	.0462	.19
LAB-10	(VOL.)	5	24.7000	.0583	5	24.6740	.0503	A	10	24.6870	.0531	.22
LAB-11	(VOL.)	5	24.3200	.0255	5	24.2060	.0439	REJECT	10	24.2630	.0690	.28
LAB-12	(VOL.)	5	24.6900	.0548	5	24.6500	.0000	A	10	24.6700	.0422	.17
LAB-13	(A.A.)	5	24.1860	.1014	5	24.3780	.1112	REJECT	10	24.2820	.1425	.59
LAB-14	(VOL.)	5	24.1680	.0653	5	24.2660	.0677	REJECT	10	24.2170	.0812	.34
LAB-15	(VOL.)	5	24.7500	.0354	5	24.7500	.0354	A	10	24.7500	.0333	.13
LAB-16	(VOL.)	5	24.3160	.0297	5	24.2820	.0497	A	10	24.2990	.0425	.18
LAB-18	(VOL.)	5	24.4200	.0274	5	24.4300	.0224	A	10	24.4250	.0242	.10
LAB-19	(VOL.)	5	24.4320	.0110	5	24.4200	.0141	A	10	24.4260	.0135	.06
LAB-20	(VOL.)	5	24.2880	.0295	5	24.2760	.0456	A	10	24.2820	.0368	.15
LAB-22	(VOL.)	5	24.4800	.0758	5	24.4700	.0758	A	10	24.4750	.0717	.29
LAB-22	(A.A.)	5	24.3300	.0837	5	24.3500	.0707	A	10	24.3400	.0738	.30
LAB-25	(VOL.)	5	24.4800	.0837	5	24.2800	.1643	REJECT	10	24.3800	.1619	.66
LAB-27	(A.A.)	5	24.6000	.0000	5	24.6600	.0894	A	10	24.6300	.0675	.27
LAB-27	(VOL.)	5	24.2760	.0767	5	24.1080	.1171	REJECT	10	24.1920	.1287	.53
LAB-27	(VOL.-P.)	5	24.3020	.0487	5	24.1420	.0823	REJECT	10	24.2220	.1057	.44
LAB-29	(VOL.)	5	24.4520	.0205	5	24.5440	.0251	REJECT	10	24.4980	.0531	.22
								TOTAL	250	24.3879	.1896	.78

*Set judged to be outlier.

TABLE 6(d)

Laboratory means, coefficients of variation, and summary of t-test on between-bottle sulphur results for RU-1

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(GRAV.)	5	22.0200	.2588	5	22.0200	.2950	A	10	22.0200	.2616	1.19
LAB- 2	(GRAV.)	5	21.5440	.0434	5	21.5200	.0400	A	10	21.5320	.0413	.19
LAB- 3	(GRAV.)	5	21.5580	.0303	5	21.5280	.0179	A	10	21.5430	.0283	.13
LAB- 4	(GRAV.)	5	21.4460	.0462	5	21.4180	.0698	A	10	21.4320	.0577	.27
LAB- 5	(GRAV.)	5	21.6340	.0336	5	21.6440	.0371	A	10	21.6390	.0338	.16
LAB- 6	(GRAV.)	5	22.1640	.0207	5	22.0920	.0249	REJECT	10	22.1280	.0437	.20
LAB- 7	(GRAV.)	5	21.5560	.0498	5	21.5620	.0680	A	10	21.5590	.0563	.26
LAB- 8	(GRAV.)	5	21.2020	.0740	5	21.2100	.0775	A	10	21.2060	.0715	.34
LAB-11	(GRAV.)	5	21.5840	.0844	5	21.6140	.0691	A	10	21.5990	.0745	.34
LAB-12	(GRAV.)	5	21.8540	.0472	5	21.7940	.0410	A	10	21.8240	.0523	.24
* LAB-12	(COMB.)	5	22.3660	.1397	5	22.0600	.2379	REJECT	10	22.2130	.2446	1.10
LAB-14	(GRAV.)	5	21.6920	.1145	5	21.5800	.1583	A	10	21.6360	.1430	.66
LAB-16	(GRAV.)	5	21.3000	.0400	5	21.3100	.0354	A	10	21.3050	.0360	.17
LAB-18	(GRAV.)	5	21.6180	.0517	5	21.6580	.0589	A	10	21.6380	.0563	.26
* LAB-19	(GRAV.)	5	22.2200	.1095	5	22.2200	.0837	A	10	22.2200	.0919	.41
* LAB-19	(COMB.)	5	22.3600	.0894	5	22.2400	.2191	A	10	22.3000	.1700	.76
LAB-22	(GRAV.)	5	21.5840	.1088	5	21.5720	.0701	A	10	21.5780	.0865	.40
* LAB-22	(COMB.)	5	21.6400	.0548	5	21.7000	.0707	A	10	21.6700	.0675	.31
LAB-25	(GRAV.)	5	21.6160	.0907	5	21.6200	.0412	A	10	21.6180	.0665	.31
LAB-27	(GRAV.)	5	21.5640	.0439	5	21.6380	.0421	REJECT	10	21.6010	.0563	.26
								TOTAL	200	21.7130	.3172	1.46

*Sets judged to be outliers.

TABLE 7

Estimation of statistical parameters for RU-1 (after rejection of outliers)

Element	No. of Labs	No. of Sets	Total No. of Results	Median, %	Mean, %	95% Confidence Limits for the Mean		Av. Within-Lab cv, %	Certification Factor
						Low, %	High, %		
Zinc	24	30	300	2.240	2.237	2.221	2.253	0.66	2.1
Copper	22	35	340	0.853	0.854	0.848	0.861	0.97	1.5
Iron	21	24	240	24.40	24.40	24.34	24.47	0.25	2.3
Sulphur	16	16	160	21.59	21.62	21.49	21.74	0.34	3.4

TABLE 8

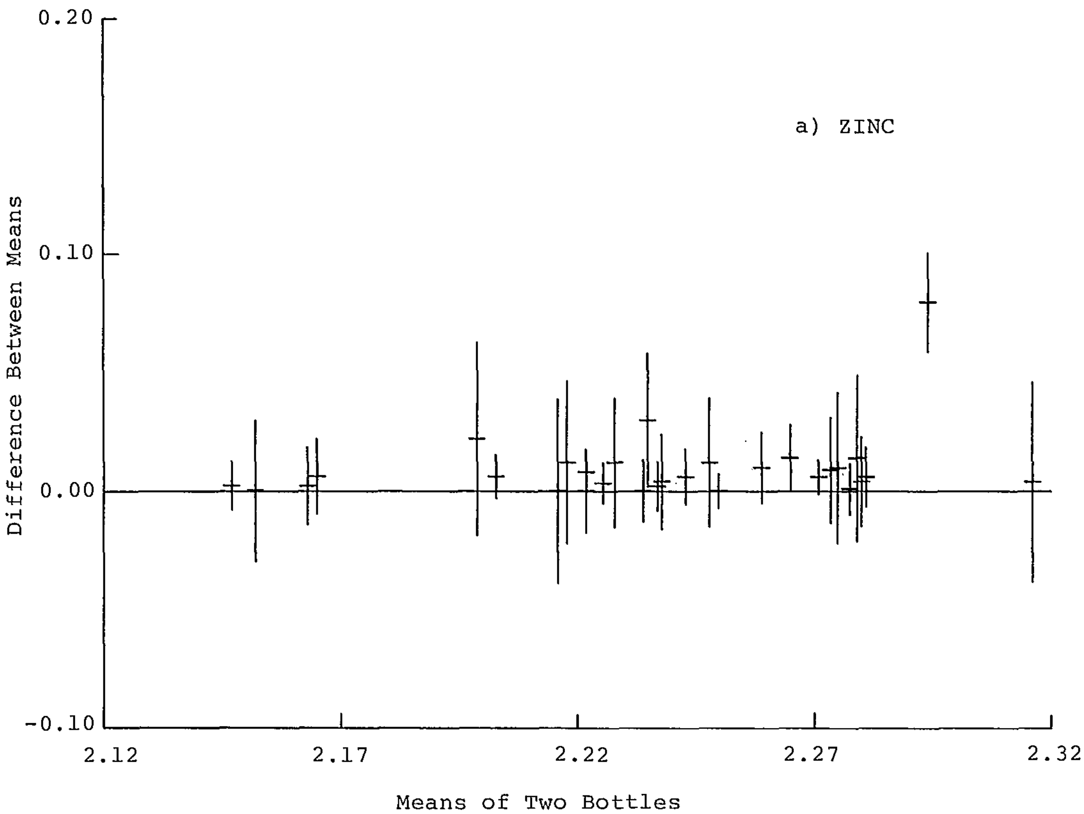
Recommended values for RU-1

	ZINC	COPPER	IRON	SULPHUR
Recommended Value, %	2.237	0.854	24.40	21.62
95% Confidence Limits				
Low, %	2.221	0.848	24.34	21.49
High, %	2.253	0.861	24.47	21.74

TABLE 9

Effect of extreme storage conditions on RU-1

Exposure Conditions			Weight Increase, mg/g	S^o Formed, mg/g	Remarks
Temp., °C	Relative Humidity, %	Time, days			
Ambient (~ 25)	--	0	--	0.8	sample directly from stock
Ambient	58	28	7.8	--	
Ambient	58	57	12.3	2.0	
Ambient	(58)	(57)	4.8	(2.0)	above sample after drying for 4 days over drierite
52	70	3	22.3	--	
52	70	7	32.8	--	
52	70	35	--	10.7	



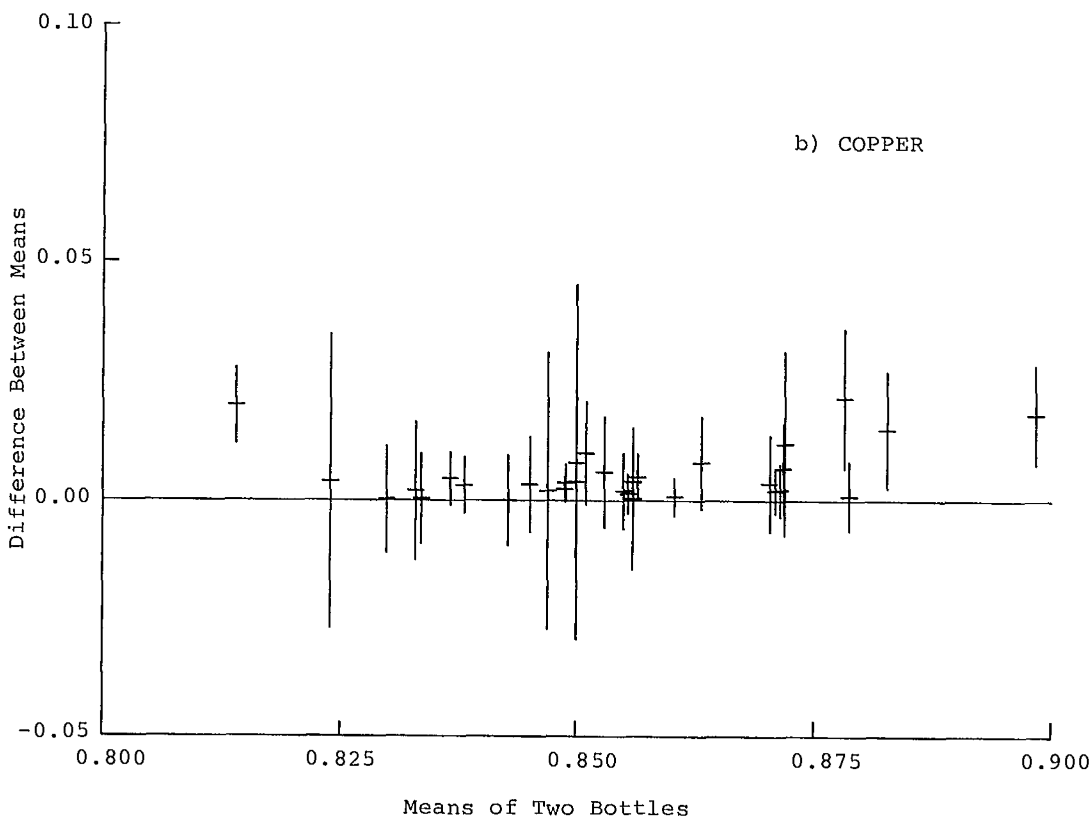
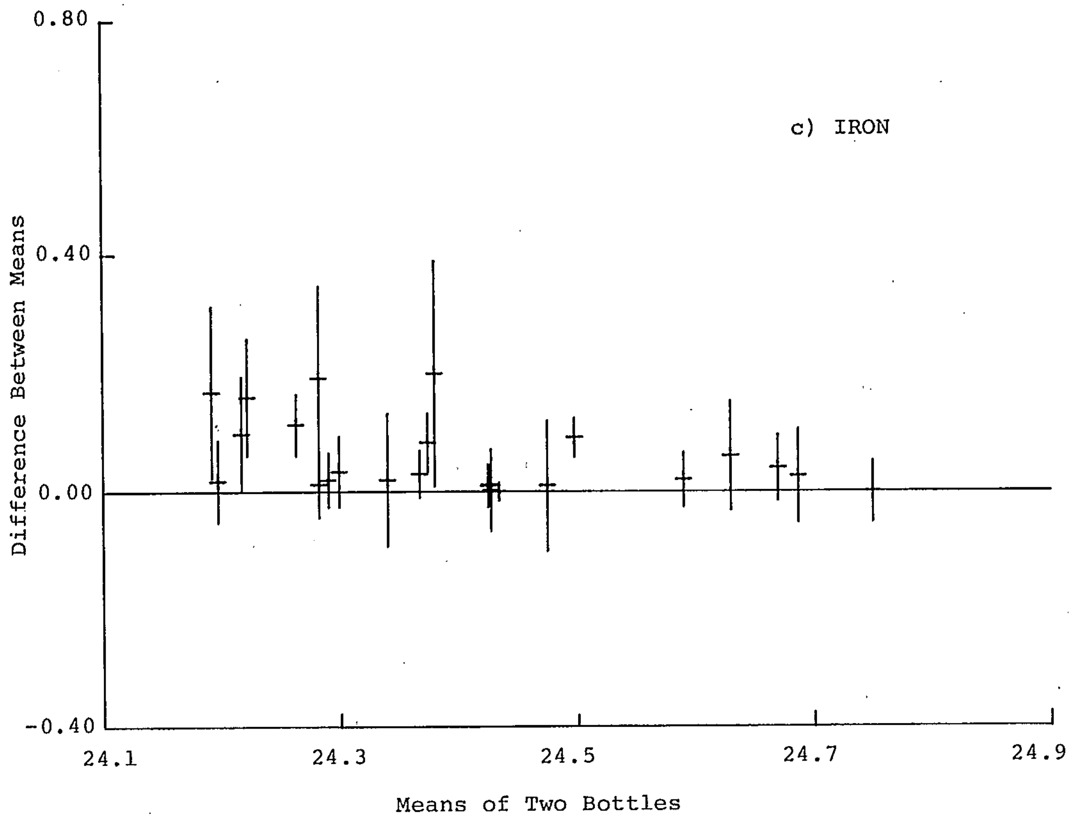
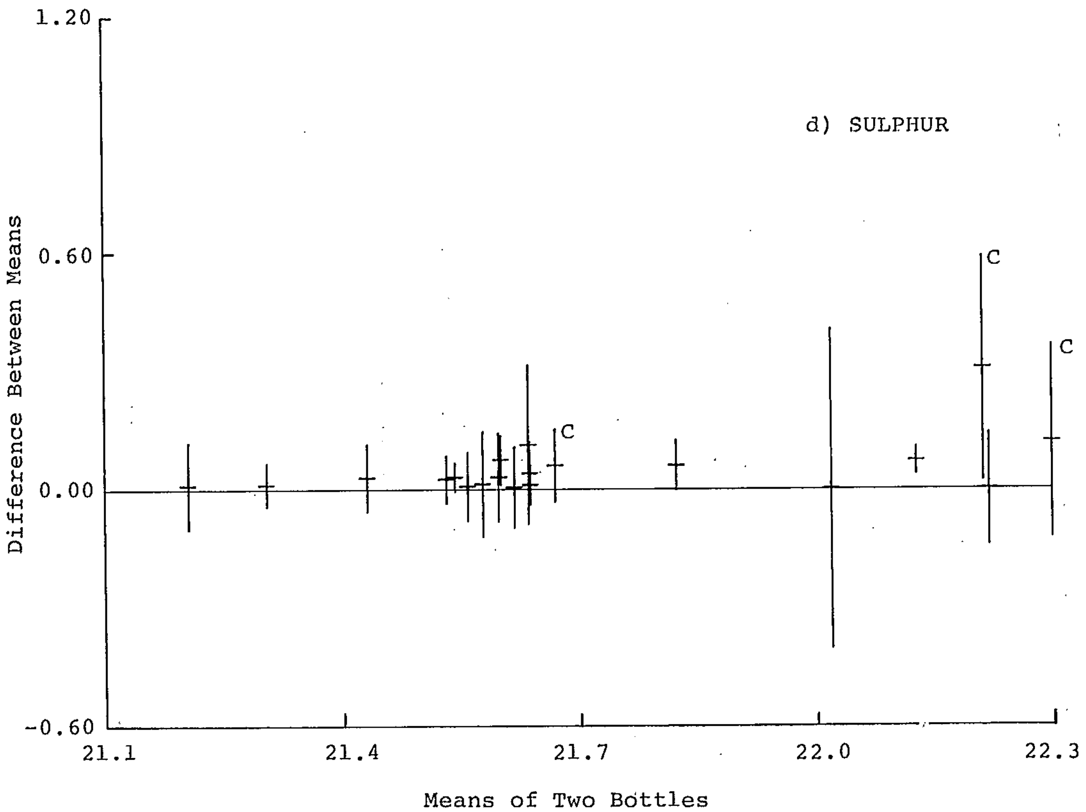


Figure 1. Illustration of degree of homogeneity



c) IRON
0.80
0.40
0.00
-0.40
Difference Between Means
24.1
24.3
24.5
24.7
24.9
Means of Two Bottles



d) SULPHUR
1.20
0.60
0.00
-0.60
Difference Between Means
C
C
C
21.1
21.4
21.7
22.0
22.3
Means of Two Bottles